Proceedings of IMECE 2004
2004 ASME International Mechanical Engineering Congress and RD&D Expo
Nov 13-19, 2004, Anaheim, California USA

IMECE2004-61320

MECHANICAL BEHAVIOR OF ELECTRODEPOSITED COPPER FILM AT ELEVATED TEMPERATURES

D. T. Read, Y.-W. Cheng, R. Geiss

Materials Reliability Division
National Institute of Standards and Technology
Boulder, Colorado 80305

ABSTRACT

The temperature dependence of the strength of a thin copper electrodeposit has been measured, by microtensile testing, from room temperature to 150 ºC. The ultimate tensile strength decreased from around 240 MPa at room temperature to just above 200 MPa at 150 ºC. The yield strength followed a similar trend. Elongation to failure increased slightly with temperature. The Young’s modulus, as measured by the unload-load slope, was well below the values expected based on averaging single-crystal elastic constants at all test temperatures. The effect of strain rate on strength at room temperature, using a range of over a decade, was low, with a weak trend upward.

INTRODUCTION

According to a widely used microelectronics industry planning document [1], junction temperatures of 150 ºC are present in ULSI devices for harsh environments, and cost-sensitive devices will have temperatures up to 85 ºC. Electrodeposited (ED) copper is the main material used as electrical interconnect in modern ULSI devices, so the mechanical properties of ED Cu at these temperatures are key inputs to reliability modeling of these structures.

An extensive set of measurements of the mechanical properties of bulk copper was reported by Carreker and Hibbard [2]. Many more detailed studies have been reported in the literature. However, the microstructures found in ED copper films are different from those found in bulk single or polycrystals [3]. Therefore, experimental values of the mechanical properties are of interest. Carreker and Hibbard emphasized that the ultimate tensile strength of copper is nearly independent of grain size, although the yield strength has the typical Hall-Petch behavior.

Methods for microtensile testing of various thin films have been developed by many researchers, although as yet no formally accepted standard test methods exist. However, measurements as a function of temperature are new. The present study is one of the first attempts at this measurement. An alternative characterization method might be nanoindentation.

MATERIALS

The ED copper specimens tested here were made in-house. Thermal evaporation was used to produce a seed layer of copper about 60 nm thick on a silicon substrate that had been deoxidized in a hydrofluoric acid solution. The tensile specimen geometry, as described below, was patterned by photolithography in the seed layer. A conventional acid-based plating solution with a proprietary brightener additive was used to plate the structures up to a thickness of 2.6 µm. The microstructure has been extensively characterized by scanning electron microscope (SEM) examination, electron back scatter diffraction (EBSD), and x-ray diffraction [3]. The lattice parameter was nominal for copper. The grain size as measured by EBSD was just over 1 µm. The preferred orientation was the typical <111> fiber texture.

TECHNIQUES

The force-probe method was used to perform the tests [4], [5]. The fixtures include a three-axis micropositioner that carries a loading pin mounted to a force sensor, and an optical microscope equipped with a digital camera. The specimen geometry is shown in Fig.1. Force readings are recorded five times per second, while images are acquired every five seconds. Stresses are calculated from the forces and the nominal specimen area, and strains are calculated from the

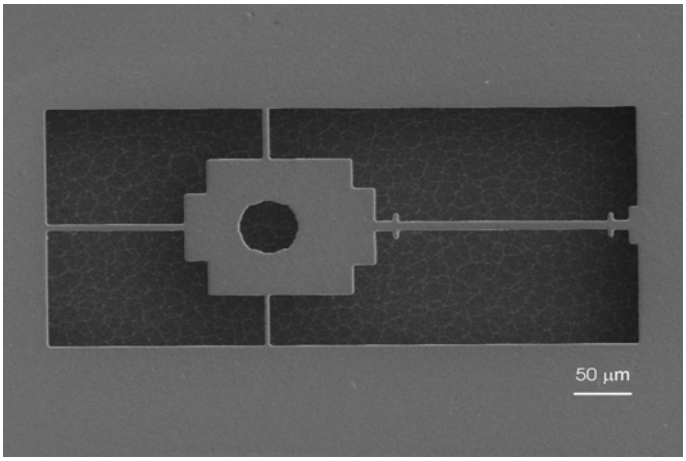

Figure 1. Microtensile specimen design used in the present study. After the tethers (three shorter strips) have been severed, the loading pin is engaged in the hole and displaced to pull the specimen to failure. The widths of the gage sections (the long strip to the right) ranged from 5 to 9 µm, and were measured individually in the SEM.

relative displacement of the two ends of the tensile section, obtained by subpixel digital image correlation.

Modifications to the previously described fixtures, needed to control the specimen temperature, were two: heater-thermocouple sets, one placed underneath the substrate carrying the films, and one on the rod holding the loading pin. Each heater was controlled by its thermocouple, through a conventional feedback controller. The temperatures reported here are the temperatures measured by the thermocouples. Tests were performed at 50, 100, and 150 ºC.

RESULTS AND DISCUSSION

Figure 2 shows a typical stress-strain curve. Table 1 lists the results. The values of Young's modulus were obtained as slopes of unloading-reloading cycles applied after initially loading the specimen into the plastic strain region. For specimens that were strained monotonically to failure, no Young's modulus values are reported, because the initial loading was typically nonlinear. Figure 3 shows a plot of the strength as a function of temperature, with literature data on pure annealed bulk copper, with a grain size of 45 µm, shown for comparison.

Table 1. Mechanical properties and sample standard deviations of thin-film electrodeposited copper: yield strength (YS), ultimate tensile strength (UTS), Young's modulus (E), and elongation to failure.

Temp., C	Number of tests	YS, MPa	UTS, MPa	E, GPa	Elong., %
25	18	234±16	257±17	72±22	2.2±0.7
50	2	213±22	229±26	NA	1.5±0.2
100	2	201±22	228±20	82±3	1.7±0.07
150	3	184±13	204±11	82±24	2.2±1.1

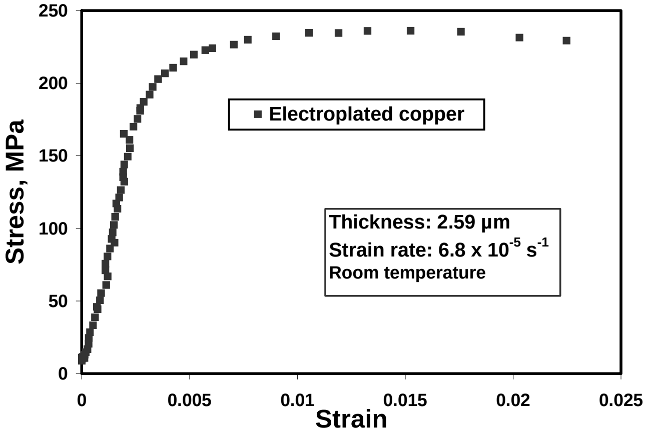

Figure 2. Stress-strain curve for microtensile specimen of electrodeposited copper at room

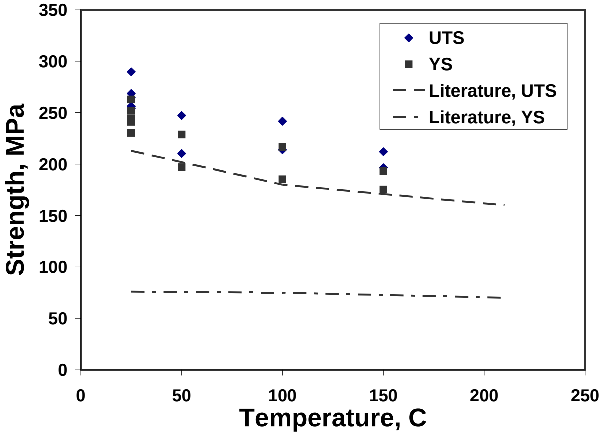

Figure 3. Yield (YS) and ultimate strength (UTS) plotted against temperature, and compared to literature values for a larger-grained material.

Figure 4 shows the strain rate dependence of the flow strength, which is minimal, with a variation over a decade less than the data scatter.

The sample standard deviations listed in Table 1 for the strengths are consistent over the temperature range, even though only the data for room temperature include a statistically acceptable number of specimens. These deviations represent mainly material variability, along with possible errors in the measurement of the widths of the specimens. The high standard deviations for the Young's modulus are traceable to the difficulty of this measurement, especially the small strain change that must be measured accurately. The high standard deviations of elongation to failure stem from the sensitivity of this parameter to small variations in specimen width or variations in microstructure at the narrowest location within the gage length.

The strength values shown in Table 1 and Fig. 1 are consistent with general trends for polycrystalline copper, considering the grain size of the present material as measured

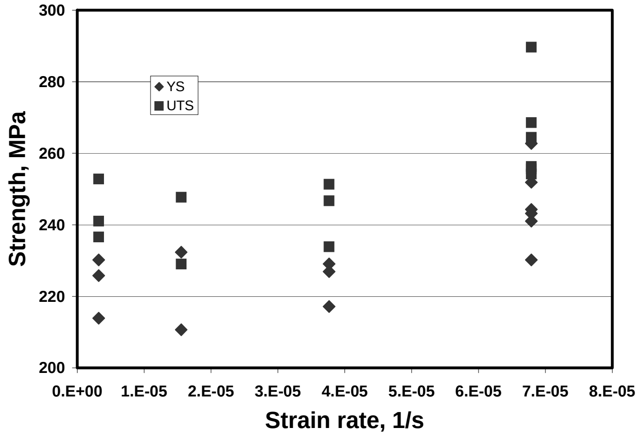

Figure 4. Yield (YS) and ultimate strength (UTS) at room temperature plotted against strain rate.

by EBSD, even though this size is much larger than the fine-scale microstructure seen in this material and reported previously [3]. We continue to investigate the relationship between the fine-scale morphological features of this material with the grain size as measured by EBSD.

The stress-strain curves showed low strain hardening, with yield strengths only slightly below ultimate strengths. Similar behavior was observed over the range of test temperatures sampled. The values of Young's modulus are low relative to the bulk polycrystalline average; a numerical modeling effort showed that this could be a result of the fine-scale microstructure [6]. The weak temperature dependence of the Young's modulus, within the scatter of the values, is consistent with the weak temperature dependence known in bulk material.

The fracture surfaces were observed using high-resolution SEM; fig. 5 shows a typical example. Figure 5 indicates a noticeable reduction of area near the fracture surface, which is consistent with significant local ductility but inconsistent with the low elongation to failure values listed in Table 1. The low elongation values are typical for thin-film tensile specimens, although a counter-example has been reported for Al [4]. A definitive interpretation of this phenomenon has not been given; the generally observed behavior is that for grain sizes 'not too small' relative to the specimen width and thickness, the imposed tensile deformation concentrates in locally-deforming regions, rather than spreading throughout the length of the gage section. Increases of strength with strain (strain hardening) and with increasing strain rate (rate effect) may contribute resistance to these strain concentrations, but as shown in Figs. 1 and 4, both strain hardening and rate effects are low in the present specimen material. So the observed low strain hardening, low rate effect and low elongation to failure are mutually consistent in the present case.

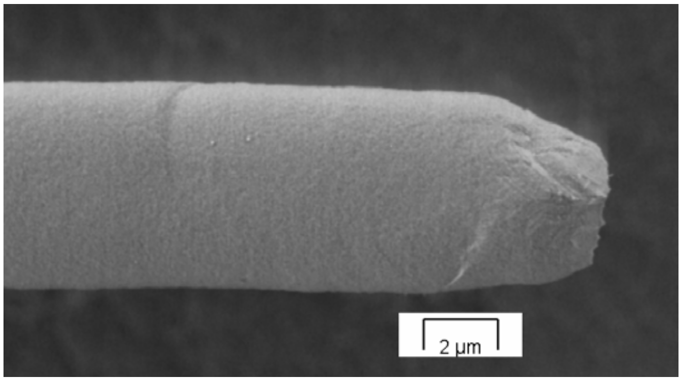

Figure 5. Typical fracture surface for the microtensile tests reported here. Note the reduction of specimen width and thickness near the fracture plane.

The mechanical property values reported here for electrodeposited copper are surprisingly similar to those found previously for electron-beam-evaporated copper [7]. In that study, the average yield and ultimate strengths were 262 and 310 MPa, respectively, and the elongation to failure was 1 %. Thicker copper electrodeposits characterized by microtensile testing had strength levels similar to the present material before annealing [8]. After annealing at 225 and 350 C, the strength decreased noticeably.

SUMMARY

The mechanical properties of electrodeposited copper were measured by microtensile testing between room temperature and 150 ºC. Strengths decreased moderately, consistent with the behavior observed for bulk Cu, while Young's modulus and elongation remained constant within the variability and uncertainty of the measurements. The yield strength was consistent with the grain size measured by EBSD, rather than the size of the fine scale morphology of this material [3]. The measured values of Young's modulus were well below the bulk polycrystalline average value. SEM observations of the fracture regions showed that the local ductility was high, even though the elongations to failure were low.

ACKNOWLEDGMENTS

Financial support from the NIST Office of Microelectronics Programs is gratefully acknowledged. Contribution of the U.S. National Institute of Standards and Technology. Not subject to copyright in the U.S.

REFERENCES

[1] International Sematech 2004.

[2] Carreker, R. P. Jr.; Hibbard, W. R. Jr. Tensile Deformation of High-Purity Copper as a Function of Temperature, Strain Rate, and Grain Size, *Acta Metallurgica* **1** 654-663, 1953.

[3] Read D.T.; Cheng, Y.-W.; Geiss, R. Morphology, Microstructure, and Mechanical Properties of a Copper Electrodeposit, *Microelectronic Materials* **75**, 63-70, 2004.

[4] Read, D. T.; Cheng, Y. W.; Keller, R. R.; McColskey, J. D. Tensile properties of free-standing aluminum thin films, *Scripta Materialia* **45** (5), 583-589, 2001.

[5] Cheng, Y.-W.; Read, D. T.; McColskey, J. D.; Wright, J. E. A Tensile-testing Technique for Micrometer-sized Free-standing Thin Films, *Thin Solid Films,* in review, 2004.

[6] Read, D. T. Atomistic Simulation of Modulus Deficit in Thin Film Copper Electrodeposits, *Journal of Applied Physics* (Submitted),2004.

[7] Read, D. T.; Dally, J. W. Mechanical behavior of aluminum and copper thin films, edited by M.A.Schen; H.Abe; E.Suhir; American Society of Mechanical Engineers: New York, 94 A.D.; pp. 41-49.

[8] Read, D. T.; Geiss, R.; Ramsey, J.; Scherban, T.; Xu, G.; Blaine, J.; Miner, B.; Emery, R. D. Nanoindentation and tensile behavior of copper films, Materials Research Society: 2003.